

RESEARCH

Toxicity Evaluation with the Microtox[®] Test to Assess the Impact of *In Situ* Oiled Shoreline Treatment Options: Natural Attenuation and Sediment Relocation

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Changes in the toxicity levels of beach sediment, nearshore water, and bottom sediment samples were monitored with the Microtox[®] Test to evaluate the two *in situ* oil spill treatment options of natural attenuation (natural recovery—no treatment) and sediment relocation (surf washing). During a series of field trials, IF-30 fuel oil was intentionally sprayed onto the surface of three mixed sediment (pebble and sand) beaches on the island of Spitsbergen, Svalbard, Norway (78°56' N, 16°45' E). At a low wave-energy site (Site 1 with a 3-km wind fetch), where oil was stranded within the zone of normal wave action, residual oil concentrations and beach sediment toxicity levels were significantly reduced by both options in less than five days. At Site 3, a higher wave-energy site with a 40-km wind fetch, oil was intentionally stranded on the beach face in the upper intertidal/supratidal zones, above the level of normal wave activity. At this site under these experimental conditions, sediment relocation was effective in accelerating the removal of the oil

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from the sediments and reducing the Microtox[®] Test toxicity response to background levels. In the untreated (natural attenuation) plot at this site, the fraction of residual oil remaining within the beach sediments after one year (70%) continued to generate a toxic response. Chemical and toxicological analyses of nearshore sediment and sediment-trap samples at both sites confirmed that oil and suspended mineral fines were effectively dispersed into the surrounding environment by the *in situ* treatments. In terms of secondary potential detrimental effects from the release of stranded oil from the beaches, the toxicity level (Microtox[®] Test) of adjacent nearshore sediment samples did not exceed the Canadian regulatory limit for dredged spoils destined for ocean disposal.

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Introduction

A series of field experiments (The Svalbard Shoreline Field Trials: Sergy *et al.*, 2003) were conducted on the beaches of Spitsbergen, Svalbard (78°56' N, 16°45' E) to assess the efficacy of the following *in situ* oil spill treatment options: natural attenuation (natural recovery), sediment relocation (surf washing), sediment mixing (tilling), and bioremediation (nutrient amendments). While these treatment options may be applied anywhere in the world and on various types of beaches and stranded oils (Sergy *et al.*, 2003), they offer an operational advantage for use in remote areas, such as the Arctic, in that no oiled materials are generated or recovered that require disposal.

Natural attenuation and sediment relocation of oiled shorelines

Natural attenuation (no treatment—recovery without intervention) is a suitable spill response option where active cleanup techniques would cause more damage than leaving the environment to recover naturally, when response techniques would not accelerate natural recovery, or when safety considerations place response personnel at risk. Natural attenuation has become a preferred response option for use on shoreline sites of limited public use that are contaminated with low concentrations of nonpersistent oil.

Sediment relocation (surf washing) involves the movement of oiled material from one section of a beach to another to accelerate the natural physical removal of oil from the sediments (Owens, 1998). Usually this action involves moving oiled sediments from a higher to a lower intertidal zone, where the physical action of waves is greater and is sustained for longer periods. In some instances, treatment involves moving oiled material that is stranded by storm waves or high spring tides to a zone that is within the normal limit of wave action. Bragg and Yang (1995) suggested that oil–mineral aggregate (OMA) formation, also called clay–oil flocculation, might have been instru-

mental in the removal of oil from low-energy shorelines in Alaska following the *Exxon Valdez* spill. They demonstrated that interactions between oil and mineral fines within intertidal sediments accelerate the transport of stranded oil in beach sediments to the water column. Subsequent studies have verified that this mechanism has occurred at other oil spill sites (Bragg & Owens, 1994; Bragg & Yang, 1995; Lee *et al.*, 1997a, 2001). Oil–mineral interactions are now believed to contribute significantly to the natural removal of stranded oil within the coastal environment and to the effectiveness of sediment relocation as a shoreline treatment option (Bragg & Owens, 1994; Owens, 1999). It has been shown that OMA formation enhances the rates of oil biodegradation (Weise & Lee, 1997; Lee *et al.*, 1997a). Thus, in addition to facilitating the transport of stranded oil in coastal sediments to the water column, OMA provides an effective mechanism for the removal of oil from the environment.

In the marine environment, OMA formation is enhanced by physical processes associated with wave-energy and turbulence that maintain mineral particles in suspension, break oil into small droplets, and increase the opportunity for collision between fine particles and oil droplets. Depending on the oil/mineral ratio of the aggregate, OMA can float (or be neutrally buoyant) and be dispersed at sea by currents, or can sink, in which case the oil becomes incorporated into marine sediments (Lee & Stoffyn-Egli, 2001).

Assessment of the environmental impacts of natural attenuation and sediment relocation is a challenging task as the treatment effects may appear in more than one compartment within the ecosystem. This type of assessment is difficult due to the fact that the success of the treatment method is based on the enhancement of residual oil dispersion by dissolution, volatilization, and physical dispersion processes associated with oil attenuation and the formation of OMA. Furthermore, the prediction of the time required for habitat recovery requires more than measurements of bulk oil loss since individual components of a crude oil, with a different capacity to induce biological effects, may not be re-

moved at the same rate. There is a concern that the application of oil treatment methods may alter the bioavailability and environmental persistence of residual oil. This paper describes an effort to address these concerns by the incorporation of a series of toxicity test protocols into the experimental design.

Experimental Design

Field sites and logistics

A series of field trials and experiments were conducted on similar mixed-sediment (pebble and sand) beaches near Sveagrava (Fig. 1) on the shores of Van Mijenfjord, Spitsbergen (78°56' N; 16°45' E) in the summers of 1997 and of 1998. Full details on criteria for site selection and the experimental design of this research program to evaluate *in situ* oil spill treatment technologies have been described by Gu nette *et al.*

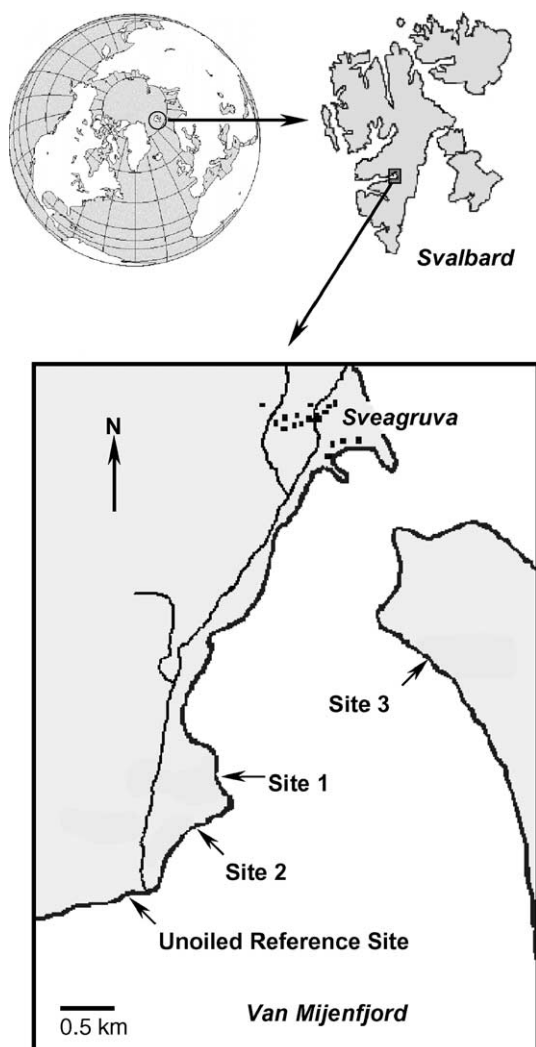


Fig. 1 Study sites near Sveagrava, Spitzbergen (Norway).

(2003) and Sergy *et al.* (2003). The selection of experimental sites was based on the presence of similar mixed-coarse sediments at the beach sites, homogeneous along-shore and across-beach sediment characteristics, the absence of surface (top 15 cm) clay deposits, continuous beach sections of sufficient length to meet experimental and sampling requirements, similar wave exposure conditions within each site, and site accessibility. Fulfillment of these criteria limited the length of overall beach surface available at each site for experimental treatments.

An intermediate oil provided by a commercial refinery, IF-30, was sprayed onto experimental plots at three beach sites (Sergy *et al.*, 2003; Prince *et al.*, 2003). Each site was exposed to different wave-energy conditions due to differences in fetch characteristics (Fig. 1). Studies on the potential for toxic effects from natural attenuation and sediment relocation (surf washing) were conducted at Site 1 (low wave exposure, 3-km wind fetch) and Site 3 (relatively high wave exposure, 40-km wind fetch). Bioremediation by nutrient enhancement and sediment mixing (tilling) was compared to the effectiveness of natural attenuation (no treatment) at Site 2 (medium wave exposure, 14-km wind fetch).

A total of 3100 l of IF-30 fuel oil was applied to Site 1 (July 30, 1997) and Site 3 (August 2, 1997) for the evaluation of natural attenuation and sediment relocation. The oil was applied with a spray boom system that distributed a 3-m swath of oil (Gu nette *et al.*, 2003). The upper intertidal zone of Site 1 was oiled below the spring high-water mark to simulate oiled shoreline conditions that would be expected at a sheltered location in the event of an accidental spill. Site 3 was exposed to considerably higher wave-energy conditions and was oiled up to the high-water berm of the swash zone, above the spring high-tide line. Predictions of sediment oil loading capacity for each site were used to guide the amount of test oil applied to each site (7.5 l/m² at Site 1; 5.0 l/m² at Site 2; and 10 l/m² at Site 3).

Full-scale treatment of plots was conducted after a period of wave and tidal exposure (5–7 days) to allow oil penetration and stabilization of the oiled zone. In this experiment, the oiled zone of two beaches (40 m length on Site 1; 80 m length on Site 3) was divided into two plots with a buffer zone between each plot (Fig. 2). One plot was designated an oiled control to evaluate the effectiveness of natural attenuation. Sediment relocation was applied to the other plot. Sediment relocation involved the excavation and relocation of sediments by a 'Bobcat' front-end loader from the oiled zone onto the lower intertidal zone (Owens *et al.*, 2003). Sediment samples were collected from the oiled zone and from the area of relocation, as well as from an intermediate zone (Fig. 2) to monitor

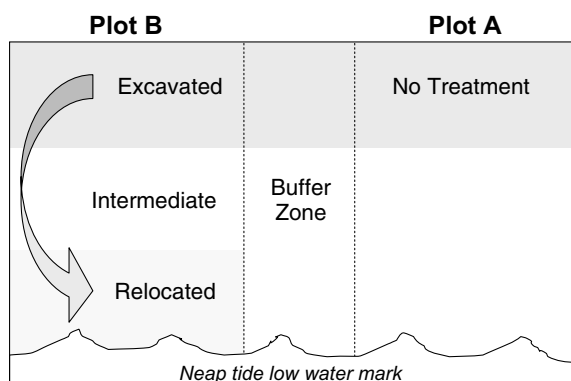


Fig. 2 Treatments and zones at Site 1. Shaded areas indicate oiled sediment. 'No treatment' is the zone of natural attenuation.

the changes that resulted from the sediment relocation action and the extent of oiled sediment transport into surrounding beach areas by natural processes (Gu nette *et al.*, 2003).

Selection of toxicity tests

Simple, sensitive, rapid, cost-effective, reproducible and practical methods are required for the assessment of toxic impacts during oil spill response operations to verify the effectiveness of the response actions in altering the concentration or composition of the residual oil within sediments. Variants of the Microtox[®] Test (a commercial biotest method accepted by regulatory agencies) were used for time-series monitoring of sediment and water toxicity in this study. This bioassay is based on the measurement of changes in light emission by a nonpathogenic bioluminescent marine bacterium (*Vibrio fischeri*) upon exposure to the test samples. The Microtox[®] Test has been used extensively worldwide over the last 18 years for toxicity screening of chemicals, effluents, water, soil and sediment, for contamination surveys and for environmental risk assessment (Cook & Wells, 1996; Bombardier & Bermingham, 1999; Pardos *et al.*, 2000; Pica-Granados *et al.*, 2000; Doherty, 2001; Guerra, 2001; Martin *et al.*, 2001; Dizer *et al.*, 2002). The application of this test for monitoring the efficacy of oil spill remediation methods has been proven (Lee *et al.*, 1995, 1997b).

Semi-permeable membrane devices (SPMDs) were used to assess changes in the bioavailability and toxicity of residual oil components following treatment. These passive *in situ* samplers, developed by Huckins *et al.* (1990) and subsequently tested and evaluated (Cooney, 1995; Hodson *et al.*, 1998), consist of a low-density polyethylene tube (wall thickness 0.80 mm) that contains purified triolein, a substance that constitutes a major fraction of the neutral lipid of fish. When immersed in water, SPMDs absorb nonionic,

organic chemicals having a $\log K_{ow} > 1$, a size < 0.1 nm and a molecular weight of about 600 or less, and possibly neutral organo-metal complexes. These characteristics correspond to that of known mixed-function oxygenase (MFO) inducing compounds, including polyaromatic hydrocarbons (PAHs). The operational advantage of SPMDs is that they can be deployed at the test site to provide an integrated sample over a period of time. This characteristic makes the assay highly advantageous for field use, such as in Svalbard, where site access is limited by logistical constraints associated with the remoteness of the site and adverse climatic conditions. In terms of ecological relevance, the diffusion of dissolved neutral organic chemicals into the triolein through the pores of the polyethylene membrane represents a measure of the potential for the diffusion of compounds across the membrane of a fish's gill. The lipid can be analyzed by traditional chemical techniques to provide a list of chemicals absorbed, their concentrations in the SPMDs, and by back calculation, their concentrations in water or sediment. During this experiment, oil from the water column and sediment interstices that became concentrated in the SPMDs was used in the Microtox[®] Test.

Sample collection

Representative samples of beach sediment were recovered to the depth of oil penetration from each test zone (up to 40 cm depth) for chemical analysis and toxicity evaluation (Gu nette *et al.*, 2003). Oil loss was estimated by gravimetric analysis of dichloromethane (DCM) extracts for total petroleum hydrocarbon (TPH) based on the application of a large-sample procedure (2–3 kg), and toxicity evaluation by the Microtox[®] Test. Samples were collected post-oiling immediately before treatment, and at Day 0, 1, 5, 10, 60 and 400 (Day 0 refers to the low tide on which treatment was initiated on each site). Each test zone was subdivided into blocks (9 at Site 1, 15 at Site 3; Gu nette *et al.*, 2003) to avoid re-sampling of the same area. Subsamples (60 cm³) of homogenized sediment for hydrocarbon analysis from each test zone were immediately frozen (–20 °C) in glass jars with Teflon liners for transport to the laboratory for toxicity analysis.

A diver collected nearshore samples from the sediment–water interface along 60–100-m transect lines perpendicular to the beach from the seaward side of the relocated sediment plots at Sites 1 and 3 in August 1997 and September 1998. These samples were frozen (–20 °C) and shipped via air-freight to the laboratory for analysis of hydrocarbons by gas chromatography-mass spectroscopy (GC-MS) and toxicity evaluation using the Microtox[®] Test.

The SPMD membranes in this study contained 1 ml of triolein. Each membrane was suspended within its own perforated aluminum cage that permitted contact with surrounding water and fine, oily sediment. The caged SPMD membranes were moored just below the surface of the beach by attachment to 0.6-m steel posts driven into the sediment. For toxicity analysis of nearshore waters at Sites 1 and 3, SPMDs were secured to the bottom of the surface buoys of sediment-traps moored 75–150 m from shore (Lee *et al.*, 2003). To monitor the impact of residual oil dispersed beyond the designated monitoring area of each test site (up to 100 m from the seaward edge of oiled plots), and for comparative purposes against another local site, SPMDs were deployed at an unoiled reference site (Fig. 1). Upon recovery, SPMDs were sealed in tins and frozen.

In 1997, sediment-traps were deployed 75–100 m offshore of Sites 1 (three times) and Site 3 (twice) to determine the potential impacts of residual oil and associated mineral fines released from the study sites. As sediment-traps could not be deployed in 1998, toxicity analyses were conducted on water samples collected from the breaker zone and near-bottom water decanted from the sediment samples collected by the diver (after a 24 h settling period). The 1997 samples from the sediment-traps were frozen (–20 °C) for storage and shipment to the laboratory for analysis. Water samples collected in 1998 were placed in insulated coolers with icepacks for air transport to the laboratory for analysis within 48 h.

Toxicity analysis

Due to differences in the test material, a number of different Microtox[®] Test protocols were used to assess the toxicity of beach sediment extracts, SPMD extracts, sediment and water samples. Although EC₅₀ values are routinely used for regulatory purposes, EC₅₀ values were converted to toxicity units (TU) × 10,000 to facilitate the direct comparison of treatment effects in graphs. A toxicity unit = 1/EC₅₀ (Microbics Corporation, 1992a). Greater toxicity is reflected by higher TU values.

Beach sediments were solvent extracted using a modification of the method described by True and Heyward (1990). Moisture content of the sediment was determined and the weight of wet sediment equivalent to 10 g dry was mixed with excess anhydrous Na₂SO₄. This mixture was extracted 3–6 times (sonicated 10 min in 15 ml DCM) and evaporated to <1 ml (Zymark[®] TurboVap Evaporator). The residue was rinsed into a 15 ml graduated centrifuge tube, evaporated to <1 ml, and made up to 1 ml with DCM. Then, 300 µl was transferred to a clean centrifuge tube

and made up to 3 ml with 95% ethanol. The mixed DCM–ethanol solution was evaporated at 50 ± 5 °C under nitrogen to <1 ml. The final solution was made up to 1 ml with ethanol. Following the “Protocol for the Basic Test Using Organic Solvent Sample Solubilization” (Microbics Corporation, 1994), 990 µl of Microtox[®] diluent and a 10-µl aliquot of the sample extract were added to a cuvette in the Microtox[®] M500 analyzer containing 1000 µl of 1% ethanol-diluent (20 ppt salinity). A series of 1:2 dilutions of the extract was made to cover a concentration range from 0 to about 7500 ppm, since preliminary tests indicated that this range generally captured the EC₅₀. The range was adjusted for any samples in which the EC₅₀ was at first obtained by software extrapolation.

For sample analysis of the hydrocarbons associated with the triolein in the SPMD's, care was taken to avoid cross-contamination. The exterior of each SPMD membrane was washed with tap water, rinsed with DCM, acetone and isopropanol, and air-dried to remove sediment particles and residual surface oil. The internal lipid content of each membrane was extracted with a 1:1 acetone–dimethylsulfoxide mixture which was used in the standard Microtox[®] Basic Test (Microbics Corporation, 1992a). TU values were divided by the exposure time.

Due to the small amount of suspended solids and fine sediments in the sediment-trap samples, the Solid Phase Test (SPT) protocol (Microbics Corporation, 1992b) was modified to use less than 0.3 g of sediment. Particulates were transferred to a pre-weighed Nucleopore[®] filter (0.4 µm) under 127 mm Hg vacuum. The filter was reweighed in a tared plastic SPT tube and corrected for the weight of the moist filter. SPT diluent (2000 µl) was added to samples consisting of ≤ 200 µg sediment and 3000 µl to samples >200 µg. The SPT tube with filter, sediment, and diluent was sonicated for 7 min. For samples consisting of ≤ 200 µg sediment, the resulting 2000 µl of sample slurry was transferred directly to a glass cuvette in the Microtox[®] model M500 Analyzer. Here, 12 test concentrations were prepared by mixing 1000 µl of the slurry with 1000 µl SPT diluent, transferring 1000 µl of this mixture to another SPT tube and adding 1000 µl SPT diluent, and so on. Three controls (1000 µl Microtox[®] diluent) were included. The standard Microtox[®] Solid Phase Test (Microbics Corporation, 1992b) was performed by inoculating the SPT tubes with 20 µl of reconstituted reagent and allowing them to incubate 30 min, after which bioluminescence was measured. For samples consisting of >200 µg sediment, the test concentrations were prepared similarly, by mixing 1500 µl slurry with 1500 µl SPT diluent. The three controls contained 1500 µl SPT diluent each. Resulting EC₅₀ values were expressed in ppm wet sediment.

The 100% Acute Toxicity Test (Microbics Corporation, 1992a) was applied to samples of breaker zone water, overlying water recovered in sediment-traps, and water in the sediment samples collected by divers along the bottom transect lines. Filtered seawater was used as diluent. The salinity of the diluent (32 ppt) was adjusted to that of the sample within ± 1 ppt by adding sodium chloride or water (Millipore[®] Super-Q) as required. Two millilitres of sample were transferred to a glass cuvette in the Microtox[®] model M500 Analyzer and the standard test concentrations of 0%, 12.4%, 24.8%, 49.5% and 99% were analyzed as specified by the test protocol.

Analysis of hydrocarbons

The concentration of TPH in the oiled sediment was monitored gravimetrically using a large sample extraction procedure (Gu nette *et al.*, 2003). Time-series changes in residual oil concentrations at Sites 1 and 3 are presented in a corresponding paper by Owens *et al.* (2003).

Changes in the composition and concentration of oil released into the coastal environment from the application of the *in situ* treatment options were monitored by GC/MS analysis. Particles were isolated from the diver-collected nearshore sediment samples by decanting the aqueous fraction after a 24-h settling period. An aliquot was taken for dry weight determination. Surrogates (*n*-dodecane, D26; hexamethylbenzene; *n*-hexadecane, D34; 9,10-dimethylanthracene; *n*-tetracosane, D50; *n*-dotriacontane, D66; *n*-hexatriacontane, D74) were added to the remaining particulate fraction which was extracted three times (sonicated 10 min with 45 ml DCM). This extract was passed through a column packed with anhydrous Na₂SO₄ into an evaporating flask and concentrated to 250 μ l (Zymark[®] TurboVap Evaporator). Surrogates were added to the water fraction of the sediment-trap samples, which was extracted in a separatory funnel by shaking three times with 25 ml of DCM for 2 min. The extract was treated as described above. Final DCM extracts from the water and particulate fractions were solvent-exchanged to hexane. A 2.0 μ l aliquot of the hexane extracts was injected into a Hewlett-Packard 5890 Series II gas chromatograph equipped with an HP 5972A mass selective detector (MSD). The GC was equipped with an HP-5 MS capillary column (30 m, 0.25 mm I.D. and 0.25 μ m film thickness) and an injection port in splitless mode. The operating conditions for the GC were: constant flow, 1 ml/min; injection port, 290 $^{\circ}$ C; transfer line, 320 $^{\circ}$ C; initial GC oven temperature, 50 $^{\circ}$ C held for 2 min; temperature ramp, 6 $^{\circ}$ C/min to 300 $^{\circ}$ C, held for 21 min; total run time, 64.7 min. The MSD was operated in selected ion

monitoring (SIM) mode for quantifying specific saturated hydrocarbons, PAHs, and sulfur heterocyclic constituents. A 300- μ l aliquot of the hexane extract was evaporated to dryness and weighed to determine the total extractable organic material. Time-series data of the ratio analyte/steranes were used to verify that IF-30 was the predominant source of residual oil in the environmental samples at the start of the experiment (approximate value of 150) and as an index of biodegradation (Wang *et al.*, 1994; Wang & Fingas, 1995).

Results and Discussion

Effectiveness of natural attenuation and sediment relocation

Sediment relocation significantly reduced the concentration of total petroleum hydrocarbons within the experimentally oiled zones at Sites 1 and 3. Details on treatment efficacy in terms of oil loss are provided in corresponding papers in this volume (Owens *et al.*, 2003; Sergy *et al.*, 2003). In comparison to natural attenuation, sediment relocation reduced oil persistence by more than a year at Site 3 where the oil was stranded in the upper intertidal and supratidal zones, above the zone of normal wave activity (Owens *et al.*, 2003). An effect of the same magnitude was not observed at Site 1. Although sediment relocation at Site 1 accelerated the short-term (first week) rate of oil loss, the same degree of oil loss was achieved by natural attenuation within 60 days. This result is attributed to the fact that the test oil was applied to this lower energy beach within an area of the intertidal zone routinely reworked by waves (Owens *et al.*, 2003). Microscopy studies confirmed that OMA formation occurred naturally at both sites and that the process of OMA formation was accelerated by the sediment relocation treatments, as this action facilitated the interaction between the stranded oil and mineral fines (Lee *et al.*, 2003).

Alterations in the toxicity of oiled beach sediments

Toxicity tests of beach sediment, nearshore sediment, and water samples recovered from the two study sites were designed to address environmental concerns associated with the release of stranded oil associated with the application of natural attenuation and sediment relocation strategies.

At Site 1, toxicity data obtained from beach sediment samples collected immediately after sediment relocation (Day 0) strongly indicate that oil applied to the upper zones of the beach was effectively removed from the application zone by the treatment action

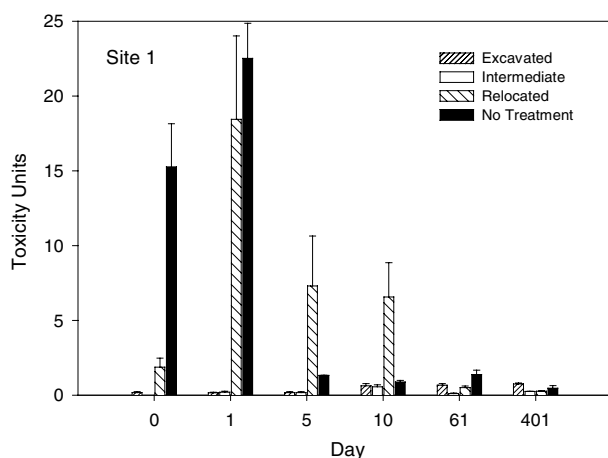


Fig. 3 Microtox[®] Test results for beach sediments at Site 1 (\pm one standard error).

(Fig. 3). Although there was little or no evidence of toxicity within the intermediate zone (Fig. 2), a high toxicity signal was recorded in sediment collected from the natural attenuation, or 'no treatment' plot. Samples recovered from the top of the 30–80-cm high pile of relocated sediment, situated at the base of the beach-face slope just above the low-water line, on Day 0 only generated a low toxicity response (Fig. 3). This observation may be attributed to capping, or mixing, of the oiled sediment with clean sediment from below the depth of oil penetration by the excavation process. After the excavation procedure, the beach profile returned to its normal slope within several tidal cycles. The relocated sediment pile was redistributed by wave action (Owens *et al.*, 2003), which resulted in the exposure of more heavily oiled sediment. This hypothetical scenario over two tidal cycles (between Days 0 and 1, Fig. 3) provides an explanation for the observed changes in mass balance of the oil applied to the beach (Owens *et al.*, 2003), as well as for the observed increase in toxicity within the relocated zone to a value similar to that of the untreated oiled plot.

Sediment toxicity at Site 1 decreased rapidly within the oiled zone (upper intertidal zone) that was designated for natural attenuation (Fig. 3) and at the same rate as total oil concentration (in less than 5 days). A similar rate of decline in sediment toxicity was not observed in the relocated sediments of the treated plot. A pronounced toxicity response remained evident in this lower intertidal zone for a 10-day period (Fig. 3). The difference in sediment toxicity between treatments may be attributed to an increase in oil persistence caused by the burial of oil, or increased bioavailability due to enhanced surface area as a result of interaction of the oil with fine mineral particles during sediment relocation. The evaporative loss of the light and most toxic fractions of the IF-30 oil may have been greater within the upper intertidal zone of the beach, con-

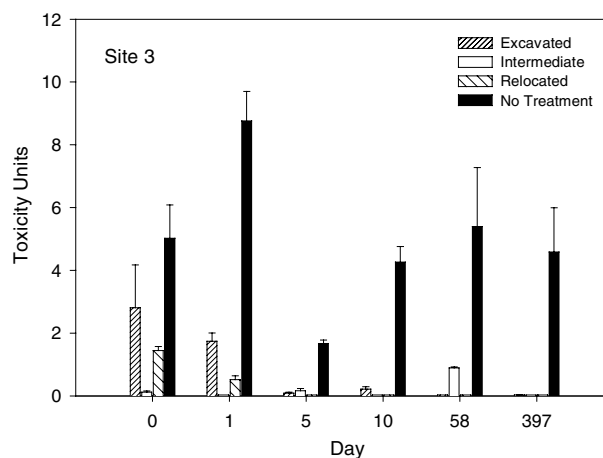


Fig. 4 Microtox[®] Test results for beach sediments at Site 3 (\pm one standard error).

tributing to the more rapid decrease in toxicity of samples collected from the no treatment zone. However, on an operational scale, by Day 61, sediment relocation treatment at Site 1 offered little advantage over natural attenuation under the scenario that was tested at this site.

At Site 3, in terms of both bulk oil removal and sediment toxicity reduction, sediment relocation was demonstrated to be a highly effective treatment option in comparison to natural attenuation (Fig. 4). This site is exposed to locally high wave-energy levels, as it has a 40-km wind fetch, and the oil was placed high on the beach immediately below the high-water berm of the swash zone and above the spring high-tide line. A significant and protracted toxic response was observed in sediment samples recovered from the untreated plot one-year later. Toxicity levels on the section of beach treated with sediment relocation were essentially reduced to background levels within 5–10 days. Significant lateral migration of oil from the untreated plot appears to have been limited by the location of the stranded oil and by the depth of penetration, with the exception of one area within the intermediate zone of the relocated plot on Day 58 (Fig. 4). On Day 397, although a toxic response was observed in the oiled zone of the natural attenuation plot, no toxicity was detected in downshore sediment samples recovered from the treated plot at locations on the lower beach-face equivalent to the intermediate and relocated zones.

Toxicity of nearshore waters and suspended sediments

Coastal waters in the immediate vicinity of the field sites (>100 m) were not toxic to the bacterium *V. fischeri*. In comparison to the controls of the Microtox[®] Test procedure, there was no significant reduction of light emission by *V. fischeri* with increasing

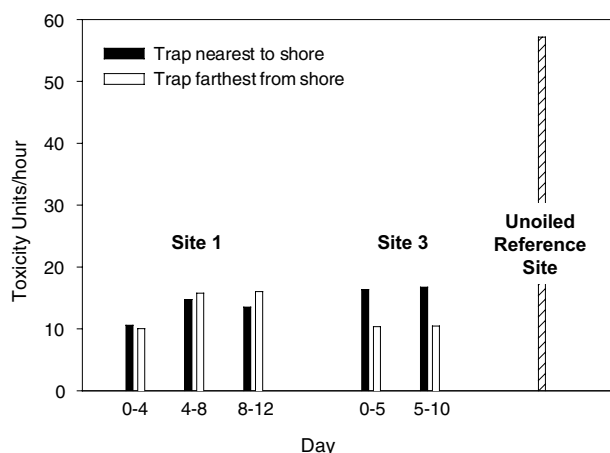


Fig. 5 Microtox® Test results for SPMDs deployed off Site 1, Site 3 and the unooled reference site.

concentration (to 99%) of water from sediment-traps, breaker zone, and nearshore bottom water. The triolein within the semi-permeable membrane devices (SPMDs) deployed in near-surface waters (~0.5 m depth) offshore from each of the designated sample sites generated a positive toxic response from the Microtox® Test procedure, due to its ability to concentrate organic contaminants. The data indicate a time-series increase in toxicity offshore from Site 1, as a result of natural attenuation and sediment relocation operations (Fig. 5). This observation agrees with hy-

drocarbon chemistry results for nearshore sediment samples collected by divers at Site 1 (Tables 1 and 2). However, on further investigation, it was noted that the toxicity values obtained at the experimental sites were significantly lower than those measured offshore from an unooled reference site (Fig. 5) that was located within 0.5 km of the Sveagruva coal loading facility (Fig. 1). These results suggest that elevated background levels of contaminants (e.g., PAHs from coal and traces of oil from marine shipping) in the Van Mijenfjord might have influenced the toxicity response for waters at the test sites. Since toxicity levels of SPMD extracts of waters adjacent to the oiled sites were not higher than those at the unooled reference site, it is suggested that natural dilution processes mitigated the adverse toxic effects of oil released from the test plots where *in situ* treatments were applied.

Regulators use EC₅₀ values, the effective concentration at which 50% inhibition occurs (thus, the lower the EC₅₀ value, the more toxic the sample), for the comparison of toxicity measurements. The Microtox® assay for suspended particulate material captured by the sediment-traps off Sites 1 and 3 produced EC₅₀ values ranging from 2390 to 8130 ppm (Table 3). Note that the EC₅₀ value of 2390 ppm for the most toxic sample approaches, but did not fall below, the 1000 ppm threshold used by regulatory agencies (Environment Canada) for contaminated dredged spoils that cannot be disposed of at sea (Tay *et al.*, 1997).

Table 1 Hydrocarbon concentrations in nearshore bottom samples from the sediment–water interface along 100 m transect lines perpendicular to shore, off Site 1 (Day 8) and Site 3 (Day 6)

Distance offshore (m)	Analyte concentration (µg/g)										
	Site 1					Site 3					
	10	20	40	60	80	100	20	40	60	80	100
Aromatics	0.24	0.85	0.87	1.64	0.98	1.68	0.14	0.11	0.17	0.27	0.50
Alkylated aromatics	0.96	3.08	2.90	5.30	3.14	5.50	0.41	0.33	0.36	0.86	1.66
Aliphatics	2.28	9.59	8.20	14.35	8.66	14.02	1.47	1.06	1.16	2.36	4.63
Total	3.48	13.51	11.98	21.29	12.78	21.20	2.02	1.50	1.70	3.49	6.80

Table 2 Hydrocarbon concentrations in nearshore bottom samples from the sediment–water interface along transect lines perpendicular to shore, off Site 1 (Day 399) and Site 3 (Day 397)

Distance offshore (m)	Analyte concentration (µg/g)										
	Site 1					Site 3					
	10	20	40	60	80	10	20	30	40	50	60
Aromatics	1.29	1.46	2.10	1.37	0.66	0.23	0.14	0.23	0.17	0.16	0.26
Alkylated aromatics	5.14	6.02	8.06	5.56	2.67	0.95	0.59	0.69	0.69	0.58	1.02
Aliphatics	19.14	23.56	29.05	22.03	10.05	4.21	3.10	3.02	3.26	3.02	4.20
Total	25.57	31.04	39.21	28.95	13.37	5.38	3.83	3.93	4.12	3.75	5.49

Table 3 Microtox[®] EC₅₀ and 95% confidence range for the sediment fraction collected in sediment-traps

Site	Trap distance offshore (m)	Day	EC ₅₀ (ppm)	95% Confidence range
1	100	5–8	6360	6160–6570
1	150	5–8	8130	6980–9470
1	100	8–12	4060	4010–4120
1	150	8–12	4600	4250–4970
3	75	0–5	2390	2210–2590
3	100	0–5	5600	4950–6330
3	75	5–10	7910	7000–8930
3	100	5–10	2390	2280–2510

Hydrocarbon concentrations and toxicity of nearshore sediments

The samples of surface sediment (~0–3 cm depth) collected in 1997 by divers following 60–100-m transect lines perpendicular to the beach from the seaward side of the relocated sediment plots at Sites 1 and 3 show that residual oil concentrations generally increased with distance from shore (Table 1). Oil concentrations within sediments at the same distance from shore were significantly higher at Site 1, where low wave-energy levels and shallower water conditions support a higher rate of sedimentation for fine particles that would likely be associated with oil.

An observed reduction in residual oil concentrations (Table 2) at each site (approximately twofold at Site 1; threefold at Site 3) along the same transect lines after a one year period is attributed to the dispersion of oil into the nearshore environment as a result of sediment relocation and natural removal processes. The Microtox[®] SPT was used to assess the potential impact of the identical sediment samples. In some cases the EC₅₀ values were not determined as toxicity was below the detection limit of the assay. Measurable EC₅₀ values of sediments from the 100 m transect lines ranged from 2580 to 5050 ppm and 9630 to 17,600 at Sites 1 and 3, respectively (Table 4). Under Canadian

Table 4 Microtox[®] dry weight EC₅₀'s and 95% confidence range for nearshore bottom samples from the sediment–water interface along 100 m transect lines perpendicular to shore, off Site 1 (Day 8) and Site 3 (Day 6)

Site	Distance offshore (m)	EC ₅₀ (ppm)	95% Confidence range (ppm)
1	10	Not detected	–
1	20	2580	1930–3460
1	40	4290	3380–5440
1	80	5050	4220–6050
1	100	2860	2240–3650
3	20	Not detected	–
3	40	Not detected	–
3	60	Not detected	–
3	80	17,600	13,500–23,000
3	100	9630	9180–10,100

Table 5 Microtox[®] dry weight EC₅₀'s and 95% confidence range for bottom samples from the sediment–water interface recovered along transect lines perpendicular to shore, off Site 1 (Day 399) and Site 3 (Day 397)

Site	Distance offshore (m)	EC ₅₀ (ppm)	95% Confidence range (ppm)
1	10	6440	5680–7290
1	20	3320	2840–3870
1	40	4090	3610–4638
1	60	3600	3280–3940
1	80	3320	938–11,700
3	10	Not detected	–
3	20	Not detected	–
3	30	Not detected	–
3	40	Not detected	–
3	50	Not detected	–
3	60	Not detected	–

regulatory guidelines for dredged sediments these sediments meet the criteria for ocean disposal (Tay *et al.*, 1997). Sites 1 and 3 were re-sampled in September 1998 to determine the changes in the toxicity of residual oil in nearshore sediments. Although the concentration of total oil had increased within the surface sediments (Table 2), in terms of toxicity, EC₅₀ values were undetectable at Site 3 and no significant increases in toxicity were observed at Site 1 (Table 5). Natural weathering processes undoubtedly reduced the toxicity of the residual oil in the sediments. Indeed, corresponding studies under this program have confirmed that toxic fractions within the test oil, such as the aromatics, were biodegraded (Prince *et al.*, 2003; Garrett *et al.*, 2003).

The toxicity of the nearshore bottom sediments to *V. fischeri*, the organism in the Microtox[®] Test, can be correlated with the total concentration of resolved analytes determined by GC-MS (Fig. 6). However, despite a measurable toxic response, none of the EC₅₀

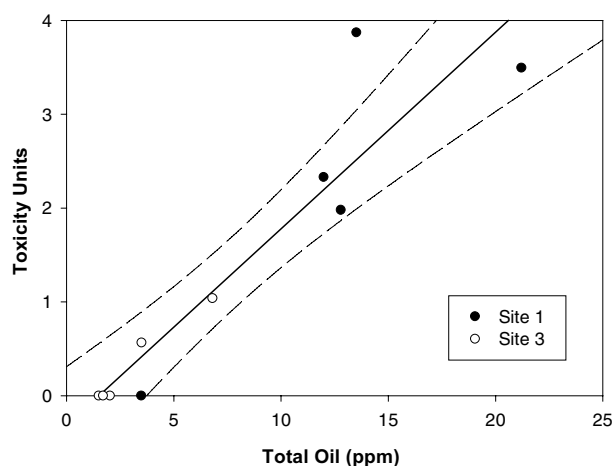
**Fig. 6** Correlation (and 95% confidence intervals) between toxicity and total oil concentration of nearshore bottom sediment samples, August 1997; $r^2 = 0.87$.

Table 6 Ratio of total resolved analytes/steranes in nearshore bottom sediment samples taken at the sediment–water interface

Sample	Distance offshore (m)					
	10	20	40	60	80	100
Site 1, Day 8	158.3	111.7	134.5	137.4	146.8	133.4
Site 1, Day 399	24.5	29.8	30.7	32.7	33.1	–
Site 3, Day 6	155.0	166.2	169.9	158.5	144.7	–
Site 3, Day 397	41.6	40.4	43.4	42.1	–	–

values obtained in this study were below 1000 ppm, a threshold value for the Microtox[®] Test set by Environment Canada for ocean disposal of dredged sediment spoils (Tay *et al.*, 1997).

Time-series data on the ratio of total analytes (sum of GC-MS resolved aromatics, alkylated aromatics and aliphatics) to the steranes, which may be considered as conserved biomarkers (Wang *et al.*, 1994; Wang & Fingas, 1995), confirm that IF-30 was the primary source oil in the samples at the beginning of the experiment (Day 3, 8), and provide strong evidence that biodegradation of residual hydrocarbons occurred within coastal sediments (Table 6). Previous OMA studies have demonstrated that oil removed from beaches into the water column in association with mineral fines is likely to be degraded to a greater extent than residual oil remaining stranded within beach sediments (Lee *et al.*, 1997a; Weise & Lee, 1997).

Conclusions

The primary objective of the Svalbard Field Trials was to evaluate the effectiveness of *in situ* shoreline treatment methods (sediment relocation, mixing, and bioremediation) to accelerate the attenuation of oil stranded on mixed sand and pebble beaches. These treatment methods were compared to natural attenuation within each site. The secondary objective was to improve our understanding of the natural processes active in these treatment actions, such as wave-induced mechanical abrasion and oil–mineral aggregate formation. New methods and techniques were developed to monitor time-series changes in bulk oil loss to address these objectives in a comprehensive manner (Guénette *et al.*, 2003; Sergy *et al.*, 2003). A corresponding paper by Owens *et al.* (2003) describes the physical loss of the stranded oil from the beaches associated with sediment relocation and mixing and by natural attenuation. Lee *et al.* (2003) highlighted the significance of oil–mineral aggregate formation on the dispersion of the stranded oil from the beaches. Prince *et al.* (2003) and Garrett *et al.* (2003) measured the effectiveness of natural attenuation and bioremedia-

tion by nutrient enrichment and/or sediment mixing to reduce residual oil concentrations. This paper, and that of Prince *et al.* (2003) about Site 2 (a medium wave-exposure beach with a 14-km wind fetch), provide information on the utility of toxicity tests (Microtox[®] Test) to identify potential detrimental secondary treatment effects and to quantify habitat recovery.

Despite the obvious difference in wave exposure between Site 1 and Site 3, the aim of this experiment was not to provide a direct comparison of results between sites exposed to different energy levels. The study team realized that this was impractical due to the large number of variables that differed between the selected study sites (e.g., location of oiling, sediment grain-size, width of the intertidal beach-face, etc.).

As with all field studies of this nature, the final experimental design is limited by costs and logistical constraints. Transportation costs and logistics for work at a remote arctic location with the adverse environmental conditions typically encountered in the Van Mijenfjord (i.e., the shore zone is only ice-free from July to December) limited the size of the field team, frequency of sampling and type of sample collected. A decision was made by the study team to evaluate the application of the experimental treatments on an “operational” scale. An attempt was made to maintain experimental parameters (mixing depth, mechanical equipment used, sites for sediment relocation, etc.) at levels similar to those that would be expected in actual response operations. The test oil was applied at concentrations that would be anticipated following an accidental spill event and at locations where it would be expected to strand on the beach. In summary, each beach within the Svalbard Shoreline Field Trial study can be considered an independent “case study” against an untreated control for the application of a specific *in situ* oil spill treatment option.

Previous studies have shown that heterogeneity within mixed-sediment beaches causes a high level of spatial variability in residual oil concentrations (Sergy *et al.*, 1991). To obtain an accurate measurement of overall oil loading within the experimental plots, a concentrated effort was made in this study to ensure that a sufficient sample density was obtained for each experimental plot to allow statistical analysis of differences between treatments at each site and over time at each site (Guénette *et al.*, 2003).

The results of experimental studies at Sites 1 and 3 demonstrated that oil removal by natural processes can be relatively rapid even in this sheltered wave-energy environment (90% of the oil was removed within the two weeks following oiling) where the oil is stranded within an intertidal zone frequently exposed to waves, but can be relatively slow where oil is

stranded higher in the upper intertidal and supratidal regions despite higher wave-energy levels. In both cases, sediment relocation accelerated these natural removal processes (Owens *et al.*, 2003).

In terms of reducing potential detrimental environmental effects, the toxicity of oiled sediments was significantly reduced in all cases where the oil was effectively removed from the beach sediments. At Site 1, the lowest wave-energy beach, sediment toxicity levels diminished rapidly (<5 days) within the oiled plots in the upper intertidal zone designated for natural attenuation. A sustained toxicity response observed for 10 days within the sediments relocated to the lower intertidal zone may have been attributed to burial of the oiled sediments and to the interactions of oil with fine mineral particles that were facilitated by wave action. The low toxicity values on Day 61 for both plots suggest that sediment relocation offers little operational advantage over natural attenuation under the environmental conditions and test scenario used at this site. At Site 3, toxicity levels of sediment samples from the beach treated by sediment relocation were essentially reduced to background levels within 5–10 days. Natural attenuation was not an effective option at this site in terms of oil reduction and the lowering of toxicity levels. One year later, oiled sediments from the untreated beach continued to generate a toxic response.

“Background” toxicity response levels could be detected by Microtox® Tests on triolein recovered from SPMDs moored with sediment-traps 75–150 m directly offshore of Sites 1 and 2 used for sediment relocation studies, and at an “unoiled” but not pristine site adjacent to the Sveagrava coal loading facility. No toxicity could be detected in the water recovered from sediment-traps. There was no evidence of long-term toxic effects; no toxicity could be detected in breaker zone water and nearshore bottom seawater samples recovered more than a year after treatment.

Studies have verified that both natural attenuation and sediment relocation resulted in the formation of oil–mineral aggregates (Lee *et al.*, 2003) that were shown to increase the concentration of residual oil within the nearshore sediments. On the basis of Environment Canada’s Microtox® Solid Phase Test protocol for evaluation of dredge spoils (Tay *et al.*, 1997), sedimentary material associated with the dispersion of OMA within the nearshore (0–150 m) environment at Sites 1 and 3 would be considered acceptable for ocean disposal.

In summary, these field trials have demonstrated the effectiveness of two *in situ* oil spill treatment options: natural attenuation (natural recovery—no treatment) and sediment relocation (surf washing). Although this one-year study has provided some evidence that residual oil concentrations may increase in nearshore

surface sediments, due to dispersion and microbial biodegradation processes, toxicity is not considered to be a potential concern within the nearshore environment as a result of their application. These results provide further support for the development and application of these *in situ* spill response options.

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